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PARTIAL TRANSLATION OF JAPANESE UNEXAMINED PATENT PUBLICATION (KOKAI) NO. 2-2856

Title of Invention: Porous Film

Publication Date: January 8, 1990

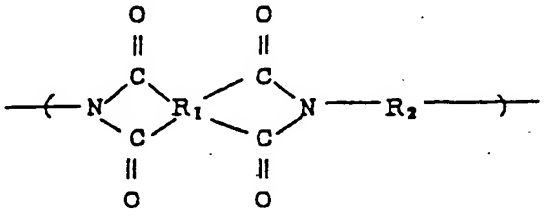
Patent Application No.: 63-212136

Filing Date: August 26, 1988

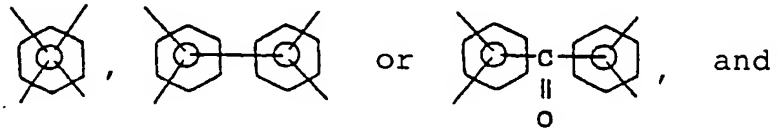
Applicant: Mitsubishi Rayon Co., Ltd.

Scope of Claim for Patent

1. A porous film for liquid filtration comprised of a polyimide having a recurring unit represented by the following general formula:

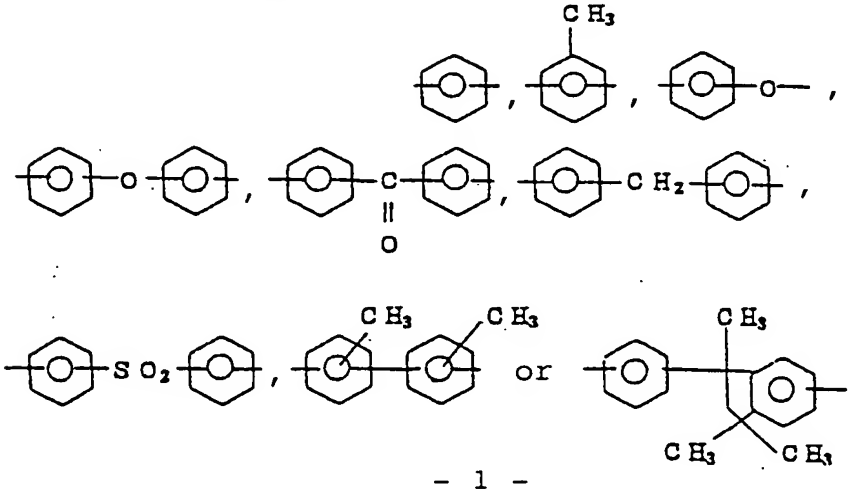


in which $R_1$ is a tetravalent group of



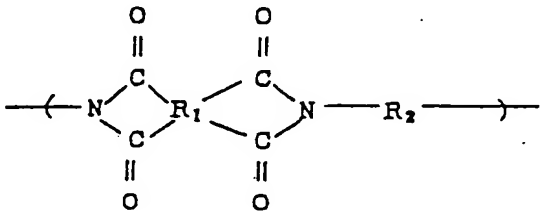
and

$R_2$ is a divalent group of

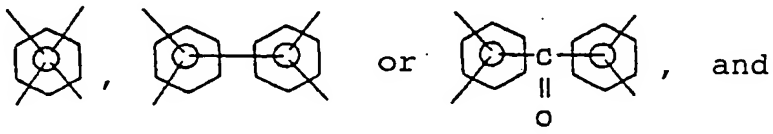


and having a mean pore size of 0.20 μm or less, a film thickness of 50 μm or greater and a water flux of 15 ml/cm$^2$·min·10 psi or greater.

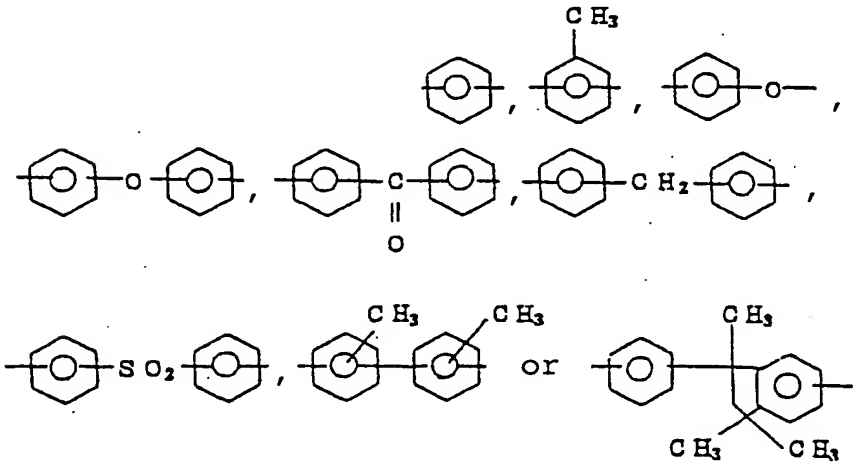
2. A porous film for gas filtration comprised of a polyimide having a recurring unit represented by the following general formula:



in which $R_1$ is a tetravalent group of



$R_2$ is a divalent group of



and having a film thickness of 50 μm or greater, a 0.11 μm particle capture efficiency of 99.9999% or greater and a pressure loss of 50 cm$H_2O$ or less at an air flow rate of 5 cm/sec.

Detailed Description of the Invention (Excerpt)

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The structure of the porous film for water filtration may not particularly be limited, but may preferably be an unsymmetrical porous film having a dense layer as the surface layer and a sparse finger-shaped or voide-like structure of large structural units in the inside. In this case, the larger the thickness of the dense layer the higher the mechanical strength, and the smaller the thickness the higher the water flux. Therefore, the thickness can be selected depending on the use, but it may usually be desirable in view of the balance thereof to be 0.1 - 20 μm, preferably 0.5 - 5 μm. The sparse portion of the larger structural units next to the dense layer may have a structure in which the spongy network structure becomes gradually large, but it is desirable to have a finger-shaped or void-like structure of high porosity in order to obtain a higher water flux.

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The pore structure of the filter is not particularly be limited, like in the porous film for water filtration, and can be, for example, an unsymmetrical structure consisting of an entirely homogeneous or heterogeneous spongy structure or of a spongy structure and a larger finger-shaped structure. The preferred pore size of the pores of the spongy structure, etc. on which the particle capture efficiency depends may vary depending on the thickness or the like, but may preferably be 0.1 - 0.5 μm. If the pore size is larger than 0.5 μm the 0.1 μm particle capture efficiency may be inferior and if the pore size is smaller than 0.1 μm the pressure loss

may undesirably be large.

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The vapor coagulation method refers to a film-forming method in which at least one surface of a thin film-like body of a polymer solution obtained by dissolving the polymer into a good solvent is contacted with a vapor containing a saturated vapor or mist of a poor solvent which is compatible to the good solvent and does not dissolve the polymer. It is considered that the vapor coagulation method makes it possible to increase, as compared with a wet film-forming method, the time between the start of the phase separation of the polymer-solvent system in the thin film-like body and the start of the subsequent polymer coagulation, thereby decreasing the coagulation rate of the polymer so that a porous film having no non-porous layer can be obtained.

The good solvent of the polymer may include N-methylpyrrolidone, dimethylacetamide, dimethyl sulfoxide, 1,4-dioxane and trichloroethylene, into which the polymer is dissolved to prepare a polymer solution.

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120 parts by weight of a polyimide resin (product of Ciba-Geigy, xu218) was dissolved into 880 parts by weight of dimethylformamide and, thereafter, casted onto a glass plate in a thickness of 254 cm using a film formation applicator to form a thin film-like body of the polymer solution. Then, to the surface of the thin film-like body, saturated steam of 2 $kg/cm^2$ was fed over 1 min. at a steam flow rate of 167 g/min from a cylindrical nozzle having a slit of a length of 30 cm and a width of 2 mm to coagulate the polymer. The thin film-like body was placed perpendicularly at a position

of 30 cm from the nozzle.

Then, the coagulated polymer was peeled off from the glass plate, washed in a water flow for about 10 min. and then dried at room temperature for 24 hours.

The film thickness, air flux, water flux, maximum pore size, mean pore size, 0.11 μm particle capture efficiency and pressure loss of the obtained film are shown in Table 1. When the film was observed by a scanning electron microscope, it was confirmed that the surface layer of the film had a dense layer comprising fine pores of a size of 1 μm or less and the dense layer had a thickness of about 15 μm. In the inside thereof, finger-shaped structures of a cylinder size of 5 - 100 μm lay in a row at an inclination of about 30° with respect to the film thickness direction.

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# PATENT ABSTRACTS OF JAPAN

(11)Publication number : 02-002856
(43)Date of publication of application : 08.01.1990

(51)Int.Cl. B01D 71/64
B01D 39/16
C08J 9/28
C08J 9/28
// C08L 79/08

(21)Application number : 63-212136
(22)Date of filing : 26.08.1988

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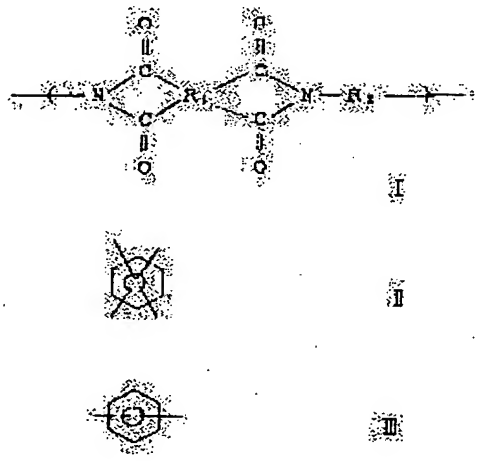
(30)Priority
Priority number : 36228678 Priority date : 13.11.1987 Priority country : JP

## (54) POROUS FILM

(57)Abstract:
PURPOSE: To obtain a porous film having excellent mechanical property, heat resistance and solvent resistance, and having a small diameter for obstructing fine particle and high fluid permeability by using polyimide having repetitive unit of specified formula and specified film properties.
CONSTITUTION: The porous film effective for the precise filtration of fine particle in liq. or gas, consists of polyimide having the repetitive unit of formula I (where, R1 = tetravalent group of formula II, etc., and R2 = bivalent group of formula III, etc.). The film has ≤0.20μm mean pore diameter, ≥50μm thickness and 15ml/cm2.min.10psi water flux. Such film has excellent mechanical property, heat resistance and solvent resistance, and has the small diameter for obstructing fine particle and high fluid permeability, because of high water flux in spite of such small pore diameter and thick thickness thereof.



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